1997-39

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Development of grazing incidence multilayer mirrors for hard X-ray focusing telescopes

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ABSTRACT

We are developing depth-graded, multilayer-coated mirrors for astrophysical hard X-ray focusing telescopes. In this paper, we discuss the primary technical challenges associated with the multilayer coatings, and report on progress to date. We have sputtered constant d-spacing and depth-graded W/Si multilayers onto 0.3–0.5 mm thick DURAN glass (AF45 and D263) and 0.4 mm thick epoxy replicated aluminum foils (ERAFs), both of which are potential mirror substrates. We have characterized the interfacial roughness, uniformity, and stress of the coatings. The average interfacial roughness of each multilayer was measured from specular reflectivity scans ($\theta_i = \theta_r$) using Cu K_α X-rays. The thin film stress was calculated from the change in curvature induced by the coating on flat glass substrates. Thickness and roughness uniformity were measured by taking specular reflectivity scans of a multilayer deposited on the inside surface of a quarter cylinder section.

We found that interfacial roughness (σ) in the multilayers was typically between 3.5 and 4.0 Å on DESAG glass, and between 4.5 and 5.0 Å on the ERAFs. Also, we found that coatings deposited on glass that has been thermally formed into a cylindrical shape performed as well as flat glass. The film stress, calculated from Stoney's equation, for a 200 layer graded multilayer was approximately 200 MPa. Our uniformity measurements show that with no baffles to alter the deposition profile on a curved optic, the layer thickness differs by ~20% between the center and the edge of the optic. Interfacial roughness, however, remained constant, around 3.6 Å, throughout the curved piece, even as the layer spacing dropped off.

Keywords: hard X-ray astrophysics, multilayers,X-ray optics

1. INTRODUCTION

Because of the excellent detection sensitivity achievable with focusing or concentrating telescopes, the development of grazing-incidence optics operating at hard X-ray energies will bring about major observational advances in this background-dominated wavelength band.

The familiar technical challenge to extending traditional grazing incidence optics into the hard X-ray band (E > 10 keV) is the decrease with energy of the graze angle for which significant reflectivity can be achieved. For a Wolter or conical approximation mirror geometry, the graze angle, θ, on a given mirror shell is related to the focal ratio by $\theta = 1/4 \times (r/f)$, where r is the mirror shell radius and f is the focal length. Extending response to high energies therefore requires either using small focal ratios or increasing the maximum graze angle over what can be

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achieved with standard metal coatings. The former can be accomplished by employing very long focal length or many small-diameter optics in the telescope design, and the latter by coating the reflective surfaces with multilayer structures, operating on the principal of Bragg reflection, that can substantially increase the maximum graze angle for which significant reflectivity is achieved over a relatively broad energy range.

We are developing hard X-ray focusing optics incorporating multilayer-coated nested mirror shells for two applications. We are currently funded under NASA's SR&T program to develop and launch the High-Energy Focusing Telescope (HEFT) balloon payload, which will operate in the 20 – ~100 keV energy band. In addition, we are participating in a technology development effort for the *HTXS* Hard X-ray Telescope (HXT). The *HTXS* mission[1] is currently being planned for a new start in 2004. The HXT instrument will extend the energy band of this observatory to E≥40 keV.

The technical approach we are pursuing for the mirror fabrication for *HEFT*, and as a possible approach for *HTXS*, is to coat depth-graded multilayers[2] on conical-approximation mirror sections similar in geometry to those developed at GSFC for *ASCA* and *ASTRO-E*,[3] and at DSRI for SODART.[4] This approach, in which the mirror shells are made in sections (usually quadrants), has the advantage over replicated Ni substrates that the optics can be inexpensively produced, have low mass, and can be coated in standard planar magnetron sputtering chambers.

In this paper, we present the results to-date of this program to develop graded multilayer-coated segmented hard X-ray optics. In Section 2, we discuss the specific technical issues associated with the fabrication of multilayers on these optics. This includes the choice of the substrate, multilayer material, multilayer quality, stress, and techniques for coating multilayers on curved surfaces. In Section 3 we present the results from the fabrication of W/Si multilayers on different potential substrates, and on a curved optic of dimensions similar to those applicable to *HEFT* and *HTXS*.

2. DESIGN AND FABRICATION ISSUES

2.1. Multilayer materials

A graded multilayer coating consists of alternating layers of high and low refractive index materials. When all of the layer pairs in a multilayer have identical thicknesses, the reflectivity peaks at angles given by the Bragg equation. To produce a broadband reflector, one uses a depth graded multilayer, where the top layers have large d-spacing to reflect the lower energies and the bottom layers have small d-spacings to reflect the higher energies. This arrangement minimizes losses due to absorption because lower energy photons travel through less material.[5]

There are several factors to consider when choosing the materials for a multilayer. The number of layers required to achieve optimum reflectivity depends on the contrast in refractive index between the materials. Since the refractive index is a function of electron density, multilayers utilizing materials with greater difference in atomic number and mass density require fewer layer pairs, and consequently, a thinner coating. This results in shorter deposition times and lower stress in the coating (which depends both on the total coating thickness and the number of layer pairs). Interfacial roughness and interdiffusion, which depend on the choice of materials, must also be minimized. Both of these effects reduce the reflectivity of the multilayer. Finally, the materials should not have absorption edges in the energy range of interest.

Possible material choices for hard X-ray telescopes, for which high quality multilayers have been fabricated, include W/Si, with a high energy cutoff at 69.5 keV (W K-edge); Pt/C, with a cutoff at 78.4 keV (Pt K-edge); and Ni/C, which has no absorption edges above 8.3 keV, and can therefore provide reflectivity up to ~100 keV. W/Si and Pt/C both require coatings with typically 50 – 200 layer pairs (depending on graze angle) to operate over the full energy range, while Ni/C coatings require approximately three times this number, due both to the smaller difference in refractive index between the materials, and to the somewhat broader energy band.

As part of the HEFT program, we are developing techniques for producing W/Si, Pt/C, and Ni/C graded multilayers on appropriate cylindrical substrates for hard X-ray telescopes. In this paper, we report the results for W/Si multilayers. Progress on the carbon-contained multilayers will be presented elsewhere.

2.2. Substrates

Grazing incidence hard X-ray telescopes use tightly nested, segmented mirror shells. The mirror substrate must be thin, to maximize collecting area, easily formable into conical sections, and be fairly stiff. In addition, the substrate must have very low surface roughness ($\leq$5 Å), since this directly affects the multilayer roughness, and hence the reflectivity. Two options that we are investigating are epoxy replicated aluminum foils (ERAFs) and a relatively new type of thin glass manufactured by the DESAG division of Schott (AF 45 and D 263). ERAFs, first developed by Serlemitsos,[3] are produced by depositing a thin film of gold onto a glass surface and bonding an aluminum foil, 0.1 – 0.5 mm thick, to the gold with epoxy. Since gold does not adhere well to glass, the gold-epoxy-aluminum foil can be easily separated from the glass, and the gold surface of the ERAF should have surface roughness comparable that of the glass. The DESAG glass, which has surface microroughness comparable to float glass, is intended for use in flat panel displays and is available in thicknesses ranging from 0.1 mm to over 1 mm. We have recently demonstrated.[6] that this glass can be thermally formed into mirror sections of the desired radius of curvature. We are primarily interested in ERAFs and DESAG glass as mirror substrates for hard X-ray telescopes, so we have investigated and judged the quality of the surfaces based on measurements of the interfacial roughness of multilayers deposited on these substrates.

2.3. Film stress

Depending on the design, hard X-ray multilayer telescopes typically require coatings with up to several hundred layer pairs that are over a micron thick. For these films, the intrinsic film stress can be a concern. For large values adhesion to the substrate becomes a problem, and at intermediate values (in the few hundred MPa range), distortion of the substrate can degrade the telescope angular resolution. This is particularly a concern for segmented optics, since the film stress will cause edge distortions that are difficult to remove in mounting. The magnitude of the effect will depend on the substrate thickness and composition.

The film stress depends on the multilayer materials, layer thickness, number of layer pairs, and the coating conditions. By varying coating chamber conditions, or by annealing of the films after coating the stress can be significantly reduced or even eliminated.

2.4. Multilayer uniformity on curved substrates

Planar magnetron sputtering systems have been widely developed for producing high-quality multilayers. Segmented mirror shells can be coated as open surfaces using planar target geometries. The advantage over cylindrical sputtering geometries is that the target-to-substrate distance can be adjusted in order to optimize reflectivity (*ie.* minimize interfacial roughness), independent of the radius of the optic. A primary concern however for depositions on curved sections is whether coatings uniform in both reflectivity and d-spacing can easily be produced using planar setups, since in the direction perpendicular to the optic axis, neither the substrate to target distance nor the angle between the substrate faces and the target are constant, as they would be for a flat sample.

Graded multilayer designs have a range of d-spacing spanning approximately an order of magnitude, so that additional variation of the d-spacing range does not have a dramatic effect on the overall broad-band reflectivity. Such variations do, however, cause the distribution of layer thicknesses to deviate from the optimum value, resulting in a decrease in average reflectivity over the band.

To quantify the effect of a non-uniform deposition rate across the mirror surface, which results in non-uniform d-spacing, we have calculated the effective area of multilayers of identical design, but with all layer-pair thicknesses varying by a constant fraction, for graded distributions intended for the HEFT balloon payload. These reflectivity curves are then averaged over the mirror surface to simulate the effect of a multilayer that has lateral grading of the d-spacing distribution in addition to the intended depth-grading. The results of these calculations are summarized in Figure 1. For a given level of nonuniformity, we minimize the loss in effective area by adjusting the deposition so that the range in coating thickness brackets the optimum thickness. From Figure 1, it can be seen that a 5% variation in deposition rate across the optic results in a $\sim$5% reduction in reflectivity.

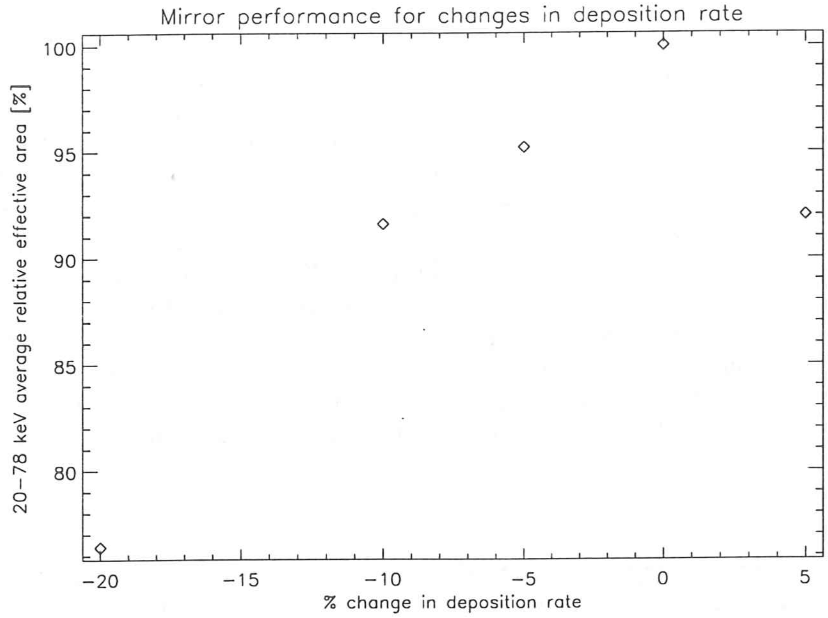

Figure 1. Relative average effective area (20 – 78 keV) for a telescope using Pt/C multilayers. At each point, the multilayer design is the same, except that each bilayer thickness is changed by the percentage indicated on the x-axis. The effective area loss due to nonuniformly coated optics would be the average loss shown here over the range in deposition rate.

3. RESULTS

3.1. Substrate and multilayer roughness

We fabricated both constant d-spacing and depth graded W/Si multilayers by magnetron sputtering at Osmic, Inc.* These chambers use planar magnetron sputtering targets. Substrates are mounted on a carousel, facing radially outward, and the targets are mounted on the sides of the cylindrical chamber. Before deposition, the chambers are pumped down to at least 2×10^{-6} Torr and then backfilled with argon to 1.5 mTorr at a flow rate of 40 cm^3/min. Power was applied to the tungsten target with a DC supply and to the silicon target with an RF supply. We ran three series of depositions at the following power settings (W/Si [Watts]): 150/1000, 240/480, 160/700. At the 150/1000 power setting, we adjusted the rotation rate of the carousel past each target in order to deposit the desired layer thickness. In this mode, we are only able to use at most two of the eight facets on the carousel. By changing the power settings to 240/480 and using a constant rotation rate for each bilayer, we were able to load samples on five facets, allowing us to coat several different substrates simultaneously. A failure in the silicon target forced us to run the last set of experiments on a different sputtering chamber, where the 160/700 power setting was used.

Interfacial roughness measurements were made by taking specular reflectivity scans with Cu K_α (8.048 keV) X-rays and comparing the results against reflectivity calculations. The number of parameters required to accurately calculate the reflectivity for a given multilayer is large, scaling as several times the number of layers. To simplify the problem, we assume that for constant d-spacing multilayers, the layer pair thickness (d), fractional metal thickness (γ), and interfacial roughness (σ) are the same for all layers, and that the densities of the materials in the film are equal to their bulk densities. In a real multilayer, deposition rates and the roughness of the film may change during fabrication, and there is no guarantee that the densities of the film are at bulk density. Consequently, with our simplifications, there will be discrepancies between the calculated and measured off-peak reflectivity. Because of the off peak discrepancies, fitting the data by minimizing χ^2 is very difficult; however, we are confident that we can determine d to within 1%, and γ and σ to within 5%.

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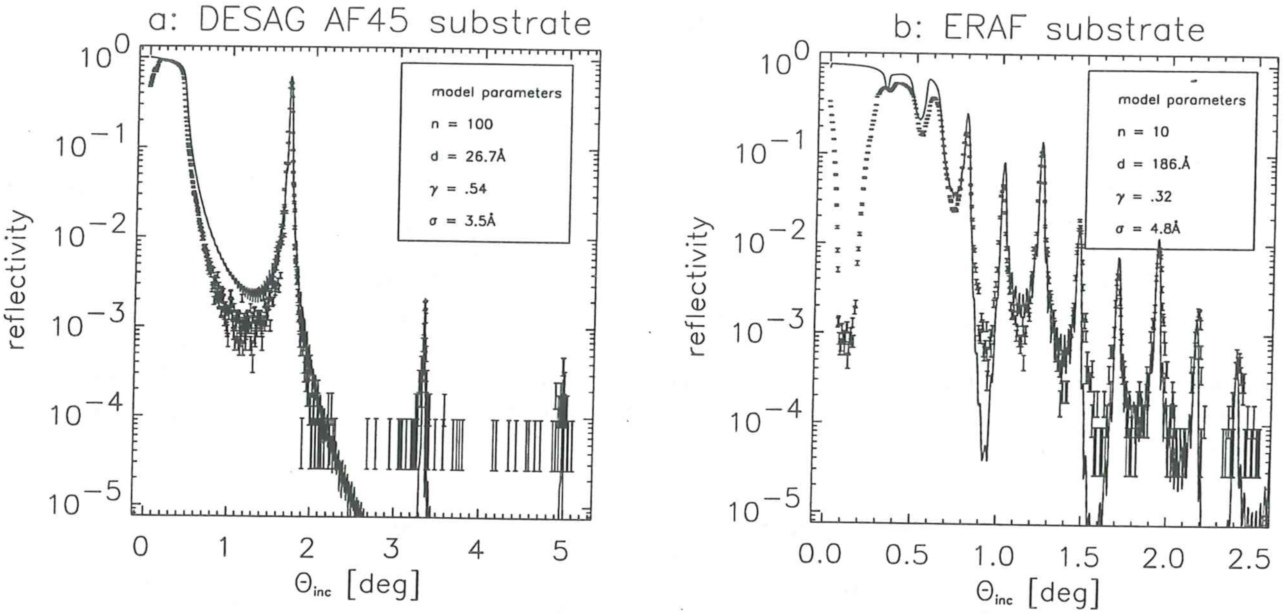

Figure 2. (a) Constant d-spacing W/Si multilayer on DESAG glass. The witness sample for this run had $\sigma = 3.1$ Å. (b) Constant d-spacing W/Si multilayer on ERAF. The roughness of the corresponding witness sample was $\sigma = 3.6$ Å. Below 0.25°, the reflectivity is reduced due to shadowing of the beam by the ERAF.

et al. 1997.[7] In summary, the film stress can be eliminated by annealing at 120° C. This temperature is low enough so that both the foil and glass substrates can be annealed after coating without compromising the surface or figure. Thus, for W/Si graded multilayer optics, the film stress can be reduced to negligible levels.

3.3. Uniformity measurements

In order to determine the magnitude of the uniformity problems associated with using planar targets to coat curved substrates, we deposited a constant d-spacing multilayer onto the inside surface of a quarter cylinder section. The substrate we used for this experiment was a 90° section of a 6.5 cm radius Schott Duran cylinder. It is important to note that no masks or baffles, other than those normally used in the chambers, were used because we are interested in the baseline characteristics of the deposition setup. We determined the thickness uniformity and variations in the interfacial roughness by taking specular reflectivity scans with Cu K_α, using the same setup as for the flat samples. Scans were taken at five positions along the curve of the cylinder: one in the center ($\theta = 0°$) and two on each side of center ($\theta = \pm 15°, \pm 30°$). A representative scan is plotted in Figure 4. The Bragg peaks of this sample are lower in reflectivity and greater in width than those of a uniformly coated mirror because the X-ray beam, which is 3 mm wide, is probing a laterally graded (*ie.* varying in thickness parallel to the surface) multilayer. We can calculate the reflectivity profile of a laterally graded multilayer by averaging the reflectivity curves of several uniform single d-spacing mirrors whose layer thicknesses cover the range of thicknesses seen on the laterally graded mirror. For each scan, we are able to extract an estimate of the range of d-spacings encountered by the beam, and the average interfacial roughness.

We did not observe any nonuniformity in the interfacial roughness, which was 3.6–3.7 Å at every point scanned. As one can see from the widths of the peaks in Figure 4, we did observe nonuniformity in the d-spacing of the multilayer. The results of the d-spacing uniformity measurements are plotted in Figure 5. One can see from the plot that there is some overlap in our estimates of the range of d-spacings at each point. Overestimating the range of d-spacings is not a concern for the uniformity measurement, because we are mainly concerned with the median layer thickness at each point. We find that the d-spacing drops off by 15–20% at 30°, which would result in a reduction of the collecting area by approximately 15%. We can easily improve the thickness uniformity by adding baffles to reduce the deposition rate along the centerline of the optic.

The results of our roughness measurements on DESAG glass and ERAF are summarized in Table 1. Because the multilayer interfacial roughness is influenced by the sputtering chamber conditions and layer-pair thicknesses as well as by the substrate roughness, in order to be able to compare results from different runs, we coat a Si wafer in each run. The constant d-spacing multilayer results in Table 1 show that DESAG glass has considerably lower roughness than the ERAF. Even with the 0.5 Å difference in roughness between the witness samples taken into account, the multilayer on the glass is still significantly smoother than the multilayer on the ERAF. These results are consistent with surface roughness measurements done on uncoated samples. Using specular scattering of Ti K_α (4.511 keV) X-rays, we measured a surface roughness of 4 Å on the ERAF. For comparison, the surface roughness of the DESAG glass is less than 3 Å.[6] The reflectivity data and model for the glass and ERAF are plotted in Figure 2. The graded multilayer results listed in Table 1 and shown in Figure 3 demonstrate that thermal forming of the DESAG glass has no effect on its surface quality. We attribute the qualitative differences between the reflectivity profiles of the flat and curved samples to the curvature of the glass and the finite width of the X-ray beam. For example, on the curved sample, the deficit in low angle reflectivity is likely due to shadowing of either the incident or reflected beam by the curvature of the sample.

Table 1. Average interfacial roughness results on candidate substrates

substrate	applied power W(DC)/Si(RF) [Watts]	d-spacing [Å]	# layers	roughness [Å]
constant d-spacing multilayers				
ERAF	240/480	185	10	4.8
Si				3.8
DESAG	240/480	27	100	3.5
Si				3.1
depth graded multilayers				
flat DESAG	160/700	25–250	200	3.5
flat DESAG	150/1000	25–250	200	4.5
curved DESAG				4.5

Our results indicate that both ERAFs and DESAG glass have sufficiently smooth surfaces to be used as substrates for multilayer mirrors. We have also shown that the thermal forming process does not change the surface quality of the DESAG glass. Other issues on the formability of these substrates are discussed in C.J. Hailey *et al.*[6]

3.2. Film stress

We have characterized the stress in graded d-spacing W/Si multilayer films with designs typical of HTXS and HEFT deposited under sputtering conditions that yielded optimal reflectivity. To determine the stress, we measured the radius of curvature of thin glass samples before and after deposition using the optical curvature measurement setup at Osmic (see Platonov *et al.* 1997[7]). Using Stoney's equation,[8] we calculate the film stress from the change in curvature radius, the thicknesses of the film and substrate, and the Young's modulus and Poisson ratio of the substrate. Table 2 shows the measured stress values.

In order to determine if the film stress can be removed either by varying deposition parameters or through annealing, we have investigated the film stress as a function of target power, and also as a function of annealing temperature for constant d-spacing multilayers. These measurements and results are presented in detail in Platonov

Table 2. Measured film stress for W/Si graded multilayers.

applied power W(DC)/Si(RF)[Watts]	# layers	thickness [μm]		radius[m]		stress [MPa]
		coating	substrate	initial	final	
160/700	200	0.625	550	-25.1	-25.7	204 ± 1
160/700	200	0.625	550	847.	368.	200 ± 2

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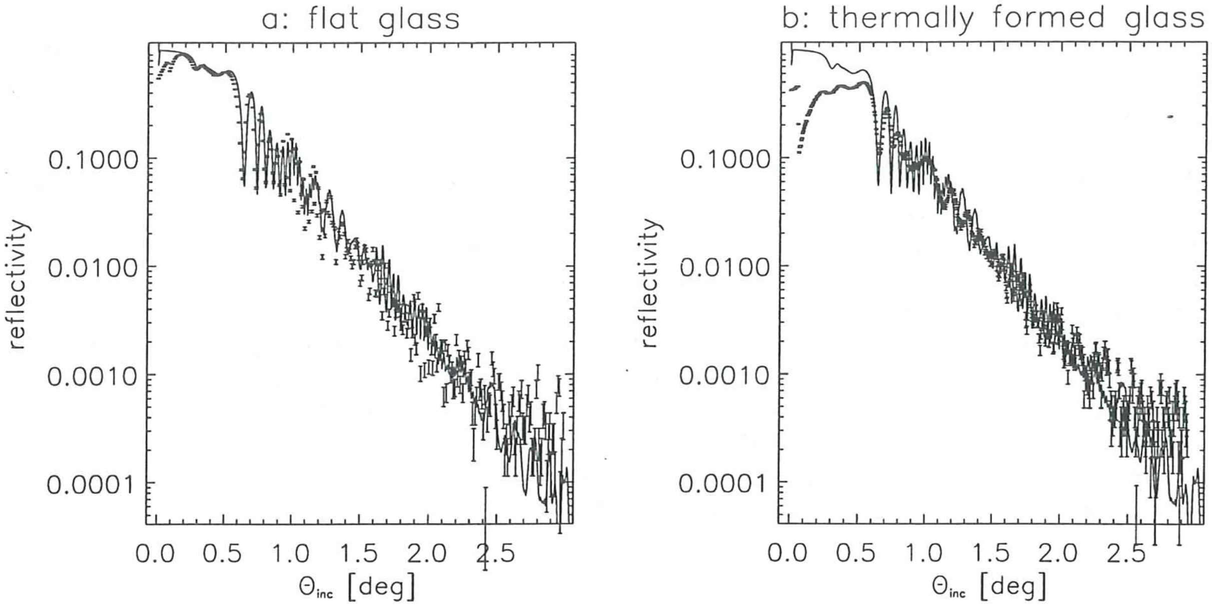

Figure 3. (a) Flat and (b) curved (10 cm radius of curvature) 2″ × 2″ DESAG glass samples. The solid line in both plots is a calculation with d(n) and γ calibrated with data from constant d-spacing samples. The coatings are identical in design: 200 layer graded multilayer with d-spacings from 25–250 Å. The only free parameter is σ, which is set to 4.5 Å in both plots. These samples were fabricated in the first series of runs with the target powers set to 150/1000.

4. CONCLUSIONS

We have established that two substrate materials, ERAFs and DESAG glass, can provide good surfaces for deposition of depth-graded multilayers. Both of these thin substrates can be formed into conic sections for segmented hard X-ray telescopes. Average interfacial roughness measurements of W/Si multilayers deposited on these substrates indicate that their surfaces have small enough microroughness, and, in the case of the ERAF, the sputtering process does not affect the epoxy film. In addition, we have determined, again using roughness measurements on W/Si multilayers, that the surface quality of the DESAG glass is not affected by the process of thermal forming into a curved optic.

With our deposition process, we have determined the stress in appropriate depth-graded multilayers, as well as the baseline thickness nonuniformity from coating a quarter cylinder section with a planar magnetron source. Improving the uniformity will not be a problem, as we can simply add baffles to reduce the deposition rate nearer to the centerline of the optic. We found no appreciable differences in the interfacial roughness among the reflectivity scans on the curved optic. The film stress in the W/Si multilayers can be removed by annealing at temperatures compatible with both ERAF and glass substrates.

ACKNOWLEDGEMENTS

We acknowledge the support of NASA's SR&T (NAGW-1919) and SBIR (NAS8-97016) programs. PHM acknowledges the support of a NASA GSRP Fellowship (NGT-50006).

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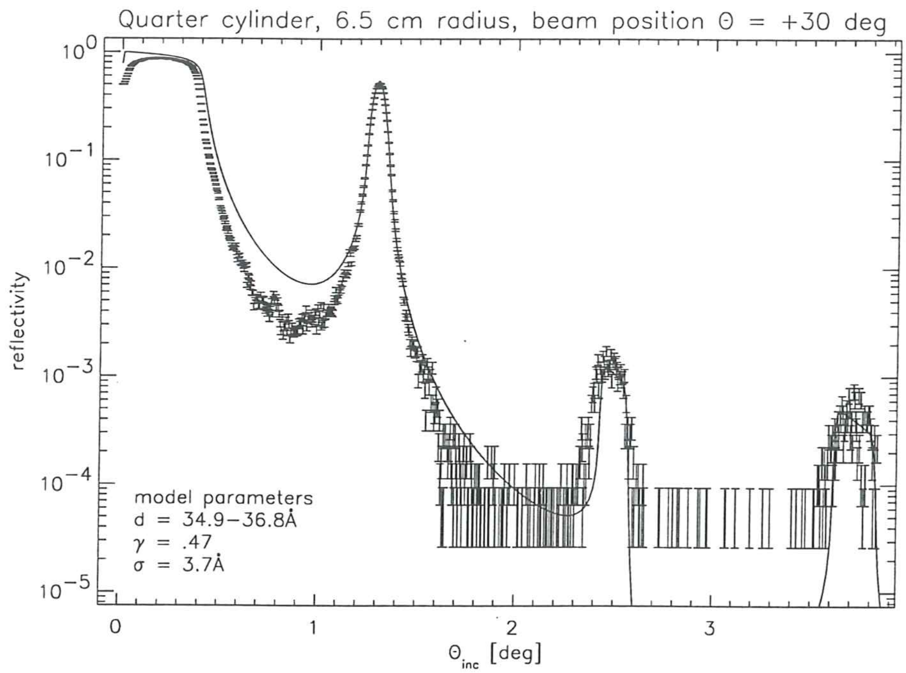

Figure 4. Data and model (solid line) of a constant d-spacing multilayer deposited on the inside surface of a quarter (90° section) cylinder. The data was taken at a point 30° from the centerline of the sample. The model is an average of 40 calculations with d-spacings linearly spaced between 34.9 and 36.8 Å. Each calculation uses $\gamma = 0.47$ and $\sigma = 3.7$ Å.

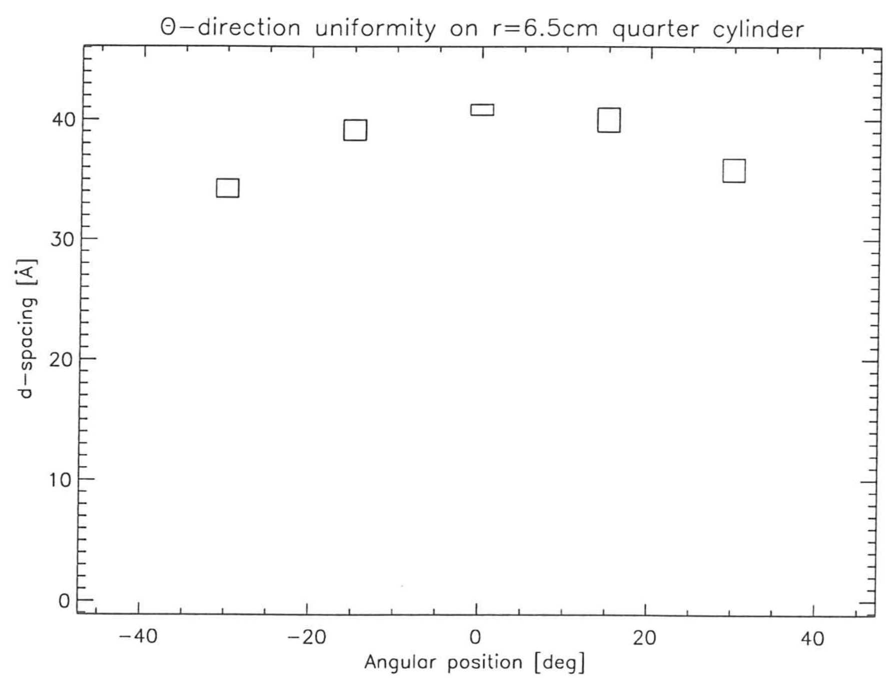

Figure 5. Uniformity measurement results. The width of each box represents the width of the X-ray beam. The height of each box corresponds to the range in d-spacings that best fit the data.